Experimental report

16/09/2018

Proposal: 9-12-510 **Council:** 4/2017

Title: Structure and dynamics of TiO2/poly(N-vinylcaprolactam) composite hydrogels

Research area: Soft condensed matter

This proposal is a new proposal

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Experimental team: Olesia TIMAEVA
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Leonardo CHIAPPISI

Samples: D2O
TiO2
poly(N-vinylcaprolactam) C8H13NO

Instrument	Requested days	Allocated days	From	To
IN15	0	0		
D11	0	1	13/04/2018	14/04/2018
IN11	8	8	05/04/2018	13/04/2018
D22	1	0		

Abstract:

Poly(N-vinylamides) are responsive polymers with a wide range of potential applications (e.g. sensors, medical materials, tissue engineering). The most studied representative of the family are the poly(N-isopropylacrylamide)-based (PNIPAM) hydrogels, but other systems, like e.g. poly(N-vinylpyrrolidone) (PVP) and poly(N-vinylcaprolactam) (PVCL) exist as well. Medical applications of PVP-based hydrogels are now wide spread due to their excellent blood compatibility, and have therefore been widely used in applications including drug delivery systems, wound dressing and tissue engineering. TiO2 containing PVCL hydrogel nanocomposites, which combine the temperature responsive properties of the hydrogel with the bactericidal and photocatalytic properties of the TiO2. Whereas a lot of information is available on PNIPAM based systems, very little is known on PVCL and PVC based TiO2-containing systems. Our aim is therefore to perform a detailed structural and dynamical study on these nanocomposites by combining SANS and neutron spin-echo spectroscopy.

Experimental report – 9-12-510
Structure and dynamics of TiO_2/poly(N-vinylcaprolactam) composite hydrogels

Experiment dates: 05/04/2018-14/04/2018
Experiment team: Olesia Timaeva, Orsolya Czakkel
Local contact: Orsolya Czakkel, Sylvain Prevost

INTRODUCTION

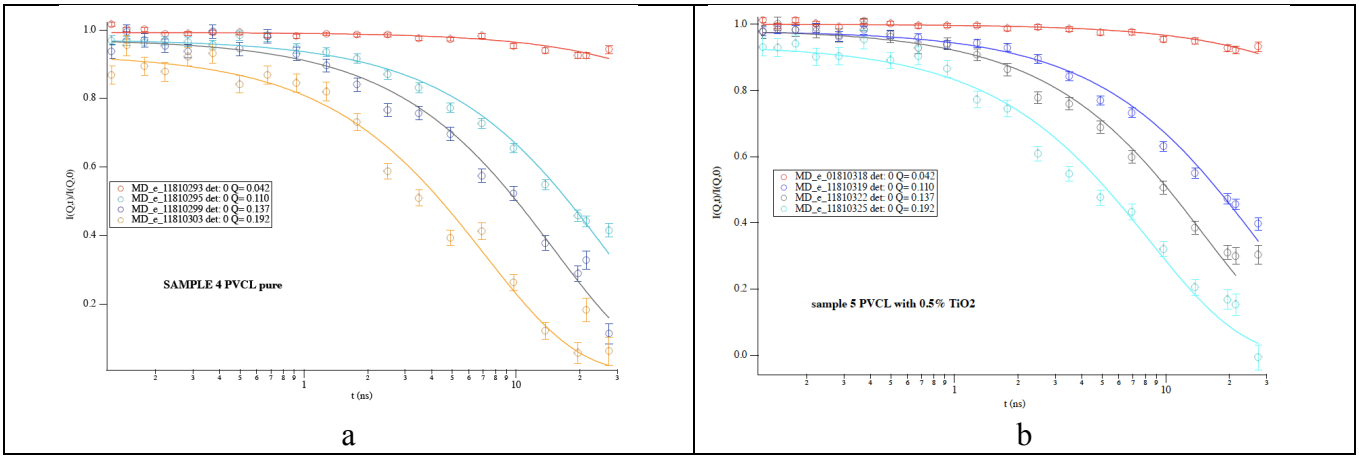
The aim is to perform a detailed structural and dynamical study on TiO_2/poly(N-vinylamides) composite hydrogels by combining SANS and neutron spin-echo spectroscopy (NSE). Poly(N-vinylamides) polymers (poly(N-vinylcaprolactam) (PVCL) and poly(N-vinylpyrrolidone) (PVP)) are responsive polymers with a wide range of potential applications. The structure of gels and their properties depend sensitively on the details of preparation and composition. These factors are not always well understood, especially in the case of organic gel – inorganic nanoparticle composites. One example of this kind of systems is TiO_2 containing PVCL hydrogel nanocomposites, which combine the temperature responsive properties of the polymer with the bactericidal and photocatalytic properties of the TiO_2.

EXPERIMENT

We investigated the dynamical behaviour of TiO_2/PVCL and TiO_2/PVP composite hydrogels (0, 0.25, 0.5 and 1 wt% of NT) at 22°C by NSE. We used the IN11 configuration with 8 Å wavelength incident neutron. Measurements were made at 5 different Q values in the range of 0.042 $Å^{-1} \leq Q \leq 0.192$ $Å^{-1}$. For SANS measurements we used the D11 small angle instrument. The incident neutron wavelength was 5 Å. Three sample-detector distances (1.4, 8 and 39 m) were used to cover the Q-range 0.016 – 5.3 nm^{-1}. Raw SANS data were corrected for the empty cell, dark counts, sample thickness and detector efficiency. The corrected scattering data were normalized by the incident beam flux to obtain the scattered intensity in absolute units.

RESULTS

It is obvious that at low q-values (q=0.042-0.137 $Å^{-1}$) the measurable Fourier time is too small to obtain the complete decay of $I(Q,\tau)/I(Q,0)$ (Figure 1). At q-value 0.192 $Å^{-1}$ the scattering functions decay to zero. For the pure PVCL and PVP and composite TiO_2/PVCL and TiO_2/PVP gels, the intermediate scattering functions appear to decay to zero at infinite time, indicative of practically ergodic behaviour, i.e. there is no frozen-in component. As the experimental intermediate scattering functions for pure and composite hydrogels do not differ much, this suggests that the presence of TiO_2 has little effect on the dynamic of polymer chains.



a b

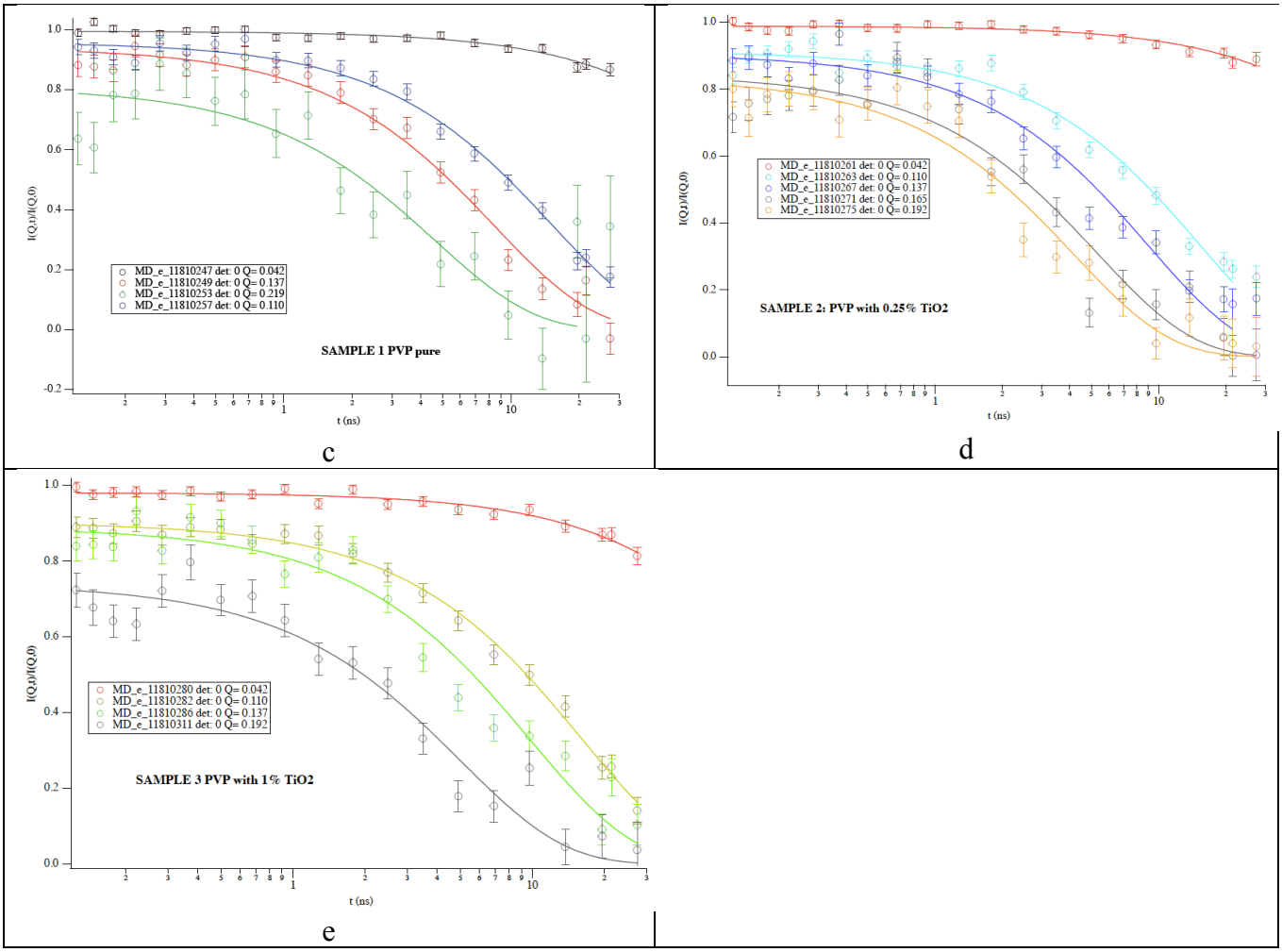


Figure 1 Experimental intermediate scattering functions from NSE with the corresponding single exponential fits for the (a) pure PVCL, (b) 0.5NT@PVCL, (c) pure PVP, (d) 0.25NT@PVP and (e) 0.5NT@PVP hydrogels.

The measured relaxation rates ($\Gamma=1/\tau$) are proportional to Q^2 (Figure 2), characteristic of diffusive motion. The diffusion coefficient (D_{diff}) is obtained directly from the linear fit to Γ vs Q^2.

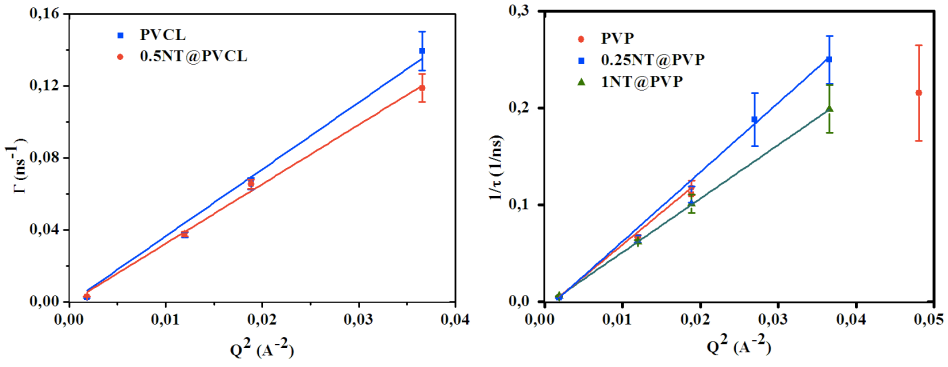


Figure 2 Relaxation rates (Γ) vs. Q^2 for (a) the PVCL and 0.5NT@PVCL and (b) the PVP, 0.25NT@PVP and 1NT@PVP hydrogels. Solid lines are linear fits.

Data analysis of the remaining part of the experiment is still under progress, but these results revealed that the hydrodynamic parameters of the polymer matrix are little affected by the incorporation of the NT.